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ABSORPTION AND FLUORESCENCE SPECTRA OF RARE-EARTH IONS IN SINGL--ETC(U)  
MAR 77 D E WORTMAN, C A MORRISON  
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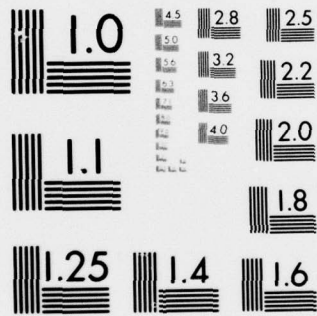


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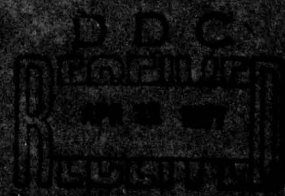


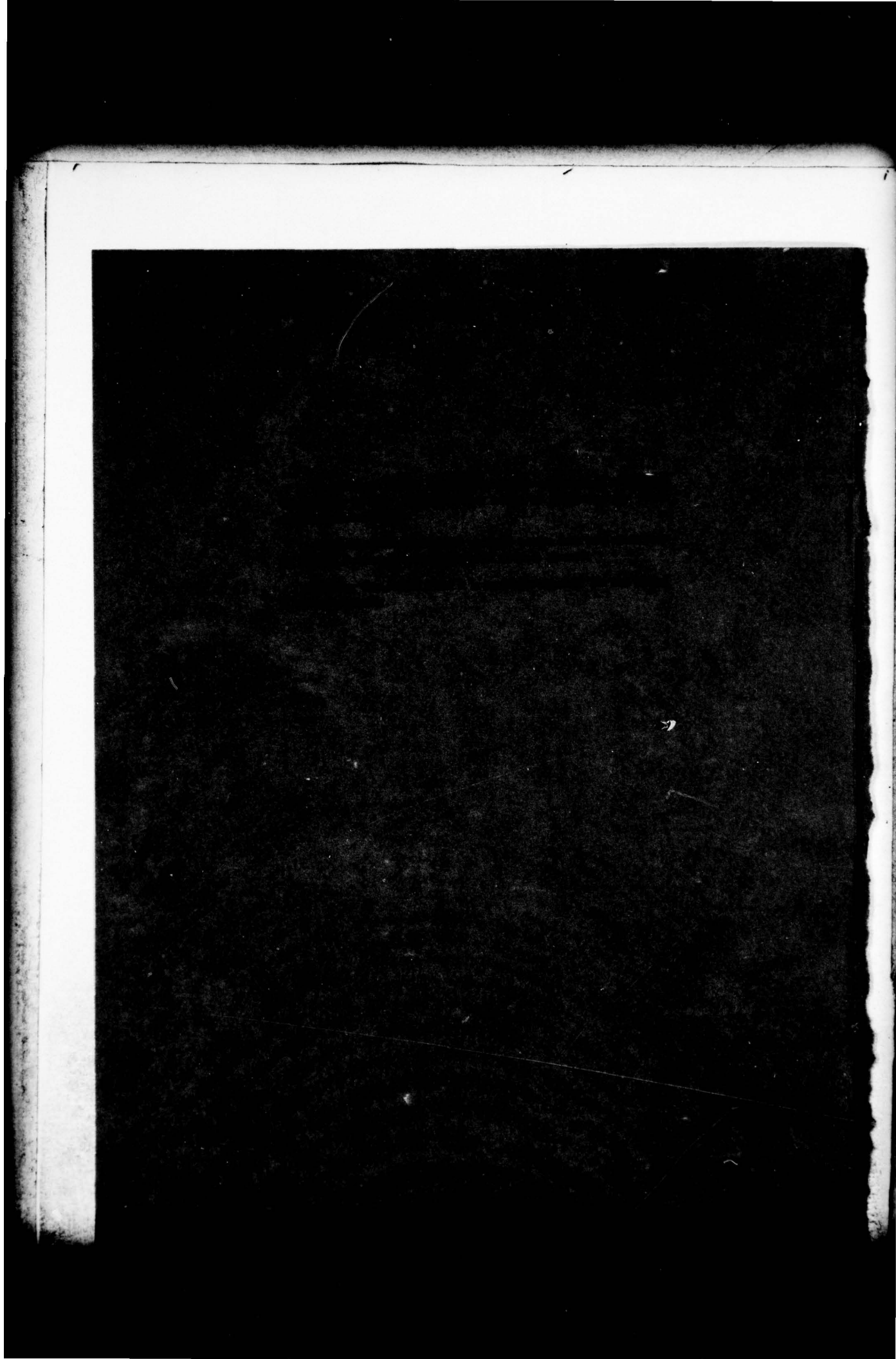
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Absorption and Fluorescence Spectra of Benzene and  
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## 1. INTRODUCTION

Several authors have reported<sup>1</sup> that single crystals containing a high concentration of the triply ionized rare earth  $\text{Er}^{3+}$  and relatively low concentrations of other rare earths are efficient laser materials. Likewise, low concentrations of  $\text{Nd}^{3+}$  in this same host has been shown<sup>2</sup> to be a low-threshold laser material. However, it has been reported<sup>3</sup> that a crystal with structure similar to  $\text{LiYF}_4$ --namely,  $\text{LiTmF}_4$  containing approximately 2-percent Nd--has extremely low emission. Several mechanisms have been described which might account for these different optical behaviors. Included<sup>4</sup> are crystal field interactions, concentration quenching, and phonon-assisted interactions. This investigation on the structurally similar single crystals  $\text{LiYbF}_4$ ,  $\text{LiTmF}_4$ , and  $\text{LiYF}_4$  containing various concentrations of  $\text{Nd}^{3+}$ ,  $\text{Tm}^{3+}$ , and  $\text{Yb}^{3+}$  was undertaken to determine the effects of these various interactions on the spectra of the various dopants. This investigation could lead to a better understanding of the effects which govern laser action in crystals.

Particular attention was given the absorption and fluorescence spectra of single-crystal  $\text{LiYbF}_4$  doubly doped with  $\text{Nd}^{3+}$  and  $\text{Tm}^{3+}$ . From these spectra, the role of the crystal field in influencing optical transitions was examined by comparing the crystal field split-ground-term energy levels of  $\text{Nd}^{3+}$  with earlier published<sup>3,5</sup> ground-term energy levels of  $\text{Nd}^{3+}$  in  $\text{LiTmF}_4$  and  $\text{LiYF}_4$ . A comparison of the fluorescence of the impurity ions in these different crystals at 85 and 300 K was made to show how energy transfer mechanisms affect luminescence.

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<sup>1</sup>E. P. Chicklis, C. S. Naiman, R. C. Folweiler, D. R. Gabbe, H. P. Jenssen, and A. Linz, *Appl. Phys. Lett.*, **19** (1971), 119; L. F. Johnson, L. G. Van Uitert, and G. E. Geusic, *Appl. Phys. Lett.*, **7** (1965), 127; L. F. Johnson, G. E. Geusic, and L. G. Van Uitert, *Appl. Phys. Lett.*, **8** (1966), 200.

<sup>2</sup>A. L. Harmer, A. Linz, D. Gabbe, L. Gillespie, G. M. Janney, and E. Sharp, *Bull. Am. Phys. Soc.*, **12** (1967), 1068.

<sup>3</sup>D. E. Wortman, S. Kulpa, and C. A. Morrison, *J. Opt. Soc. Am.*, **62** (1972), 604.

<sup>4</sup>G. H. Dieke, *Spectra and Energy Levels of Rare Earth Ions in Crystals*, Interscience Publishers, New York (1968); L. G. Van Uitert and L. F. Johnson, *J. Chem. Phys.*, **44** (1966), 3514; J. R. O'Connor, *Trans. Met. Soc. AIME*, **239** (1967), 362.

<sup>5</sup>D. E. Wortman, *J. Phys. Chem. Solids*, **33** (1972), 311.

## 2. PROCEDURE AND RESULTS

All the crystals used were grown by the "top-seeded" method.<sup>6</sup> The crystal of  $\text{LiYbF}_4$  doped with Tm was grown from a melt containing 0.5 at.%  $\text{Tm}^{3+}$ . The double-doped crystals of  $\text{LiYbF}_4$  were grown from this same mixture with 0.5 at.%  $\text{Nd}^{3+}$  being added to the melt. Each of these crystals was colorless; the crystal samples were cut without attention to orientation. The axes of one of the  $\text{LiTmF}_4$  crystals were not determined either, and it was a light-greenish yellow. The  $\text{LiYF}_4$  crystals grown from melts containing 2 at.% each of  $\text{Nd}^{3+}$  and  $\text{Yb}^{3+}$  and a  $\text{LiTmF}_4$  crystal grown from a melt containing 2.0 at.%  $\text{Nd}^{3+}$  were colorless, and crystal axes were determined for each of these crystals by x-ray techniques. These three crystals were furnished by A. Linz of the Massachusetts Institute of Technology Crystal Laboratories. All the crystals have the scheelite structure, and the rare earth is expected to substitute at the site designated X in the crystal  $\text{LiXF}_4$  where the point group symmetry is  $S_4$ .

The absorption spectra of  $\text{Nd}^{3+}$  and  $\text{Tm}^{3+}$  in single-crystal  $\text{LiYbF}_4$  at 25, 85, and 300 K were recorded over the wavelength range from 0.1875 to 2.6  $\mu\text{m}$  with a Cary 14 spectrophotometer. Absorption corresponding to transitions from the ground multiplet to all the energy levels below the band edge, near 0.21  $\mu\text{m}$ , were observed for  $\text{Tm}^{3+}$  (transitions to all except the  $^1S_0$ ). The center positions of the  $\text{Tm}^{3+}$  absorption lines are at approximately 1.68, 1.19, 0.785, 0.875, 0.66, 0.47, 0.3575, 0.299, 0.288, 0.275, and 0.2625  $\mu\text{m}$ , corresponding to transitions from the ground  $^3H_6$  multiplet to the  $^3F_4$ ,  $^3H_5$ ,  $^3H_4$ ,  $^3F_3$ ,  $^3F_2$ ,  $^1G_4$ ,  $^1D_2$ ,  $^1I_6$ ,  $^3P_0$ ,  $^3P_1$ , and  $^3P_2$  multiplets, respectively. The relative intensities of the strongest line to each  $\text{Tm}^{3+}$  multiplet, in absorbance units, in that same order are 5.5, 4.5, 4.0, 3.0, 0.5, 0.75, 2.0, 0.5, 0.6, 0.75, and 1.5. Owing to the many low-lying energy levels of  $\text{Tm}^{3+}$  which complicate the  $\text{Tm}^{3+}$  absorption spectrum, no detailed theoretical analysis was made of these  $\text{Tm}^{3+}$  data.

The energy levels of the  $^4I$   $\text{Nd}^{3+}$  ground term, using the  $\text{LiYbF}_4$  crystal, were determined from which crystal field parameters describing the electrostatic field at the impurity ion site might be obtained as was done<sup>5</sup> for  $\text{Nd}^{3+}$  in  $\text{LiYF}_4$ . The wavelength positions of the lines are in close agreement with  $^4I$   $\text{Nd}^{3+}$  absorption lines<sup>3,5</sup> in  $\text{LiYF}_4$  and  $\text{LiTmF}_4$ , but the intensities are somewhat different for certain lines as given in table I. Whether this difference is due to inversion of these certain lines owing to quite different crystal field parameters

<sup>3</sup>D. E. Wortman, S. Kulpa, and C. A. Morrison, *J. Opt. Soc. Am.*, **62** (1972), 604.

<sup>5</sup>D. E. Wortman, *J. Phys. Chem. Solids*, **33** (1972), 311.

<sup>6</sup>D. Gabbe and A. L. Harmer, *J. Crystal Growth*, **3** and **4** (1968), 544.



TABLE I. Relative heights (approximate intensities) of absorption lines used to establish given energy levels for  $\text{Nd}^{3+}$  in  $\text{LiYbF}_4$ ,  $\text{LiYF}_4$ , and  $\text{LiTmF}_4$  at 25 K.<sup>a</sup>

J-multiplet and $S_u$ -symmetry		$\text{LiYF}_4:2\% \text{Nd}^{3+}$		$\text{LiTmF}_4:2\% \text{Nd}^{3+}$		$\text{LiYbF}_4:0.5\% \text{Nd}^{3+} + 0.5\% \text{Tm}^{3+}$	
Property	$\Gamma_{k,m}$	Relative intensity	Energy ( $\text{cm}^{-1}$ )	Relative intensity	Energy ( $\text{cm}^{-1}$ )	Relative intensity	Energy ( $\text{cm}^{-1}$ )
$4F_{3/2}$	7,8	6	11,538	20	11,535	10	11,541
	5,6	55	11,597	85	11,594	30	11,600
$4I_{9/2}$	7,8		0		0		0
	7,8		132		131		132
	5,6		182		178		180
	5,6		249		246		
	7,8		528		531		
$4I_{13/2}$	5,6	20	3,948	35	3,949	21	3,950
	7,8	0.5	3,976	2	3,979	0.02	3,980
	5,6	64	3,995	83	3,997	18	3,998
	7,8	4	4,026	3	4,028	7	4,027
	5,6	24	4,205	11	4,212	0.7	4,222
	5,6	0.5	4,228	5	4,220	5.8	4,256
	7,8	10	4,238	5	(4,249)	(not resolved)	(not resolved)
$4I_{15/2}$	5,6	23	5,851				
	5,6	12	5,912				
	7,8	55	5,947				
	7,8	0.25	6,026				
	7,8	1	6,315			1.0	6,327
	5,6	12	6,347	4	6,353	1.5	6,366
	7,8	0.04	6,388				
	5,6	5	6,432	4	6,444	1.5	6,455

<sup>a</sup>To compare relative intensities, the heights of absorption lines are obtained for a particular crystal. Dopant levels and optical path lengths differ for the various host materials. Also, these relative heights are given only for transitions from the ground state since several different transitions from the remaining  $4I_{9/2}$  energy levels to known levels were used to obtain these states of the ground multiplet. The symmetry properties are those reported for  $\text{Nd}^{3+}$  in  $\text{LiYF}_4$  and  $\text{LiTmF}_4$ .

or whether these intensity differences do exist for corresponding transitions could not be determined with the present unoriented crystals. Because of the similarity in the positions<sup>3,5,7</sup> of these lines with those of  $\text{Nd}^{3+}$  in  $\text{CaWO}_4$ ,  $\text{LiYF}_4$ , and  $\text{LiTmF}_4$ , it is probable that the main difference is intensity. Therefore, the energy levels in table I are labeled consistently with the symmetry properties of  $\text{Nd}^{3+}$  in the other host crystals. The  ${}^4\text{F}_{3/2}$  energy levels which are useful in establishing the  ${}^4\text{I}_{11/2}$  energy levels by way of fluorescence measurements also are given in table I. In addition, transitions were observed from the ground multiplet to all  $\text{Nd}^{3+}$  multiplets below the band edge.

Fluorescence from the  $\text{LiYbF}_4:(\text{Nd}^{3+} \text{ and } \text{Tm}^{3+})$  at 85 and 300 K was recorded over the wavelength range from 0.6 to 2.6  $\mu\text{m}$  by using the Cary 14 spectrophotometer in the single-beam mode of operation with the linear slide wire. A 100-W Xe lamp focused on the crystal provided the energy for exciting the fluorescence. Fluorescence lines corresponding to  ${}^4\text{F}_{3/2}$  to  ${}^4\text{I}_{11/2}$   $\text{Nd}^{3+}$  transitions were not observed even with a careful inspection near 1.06  $\mu\text{m}$ , where the usually prominent  $\text{Nd}^{3+}$  emission occurs. Emission was recorded corresponding to  $\text{Tm}^{3+}$  and  $\text{Yb}^{3+}$  transitions, however, and this is described below.

Since  $\text{Nd}^{3+}$  appears to be transferring energy perhaps to the  $\text{Yb}^{3+}$  ion in  $\text{LiYbF}_4:(\text{Nd}^{3+} \text{ and } \text{Tm}^{3+})$ , the emissions from a number of other crystals at 85 and 300 K also were recorded to examine whether similar energy transfer mechanisms occur. The same experimental equipment and procedures described above were used. The fluorescence obtained for the different crystals are given in table II.

### 3: DISCUSSION OF RESULTS

As obtained from table II, the probability of transfer of excitation energy from  $\text{Tm}^{3+}$  to  $\text{Yb}^{3+}$  or from  $\text{Nd}^{3+}$  to  $\text{Yb}^{3+}$  is larger than the probability of quenching interactions between the  $\text{Tm}^{3+}$  ions. The comparison of  $\text{LiTmF}_4:\text{Nd}^{3+}$  and "pure"  $\text{LiTmF}_4$  shows that  $\text{Nd}^{3+}$  and  $\text{Tm}^{3+}$  mutually quench. A comparison of  $\text{Yb}^{3+}$  and  $\text{Nd}^{3+}$  in the same host,  $\text{LiYF}_4$ , shows that  $\text{Yb}^{3+}$  emission near 1.0  $\mu\text{m}$  and  $\text{Nd}^{3+}$  emission near 1.06  $\mu\text{m}$  diminish slightly at a lower temperature. The peak of the emission of  $\text{Yb}^{3+}$  in  $\text{LiYF}_4$  at 0.996  $\mu\text{m}$  does not appear resolved in the emission of  $\text{Yb}^{3+}$  in  $\text{LiYbF}_4$ . That this 0.996- $\mu\text{m}$  peak in  $\text{LiYF}_4$  increases in amplitude at a lower temperature indicates possibly the thermal depopulation of the  $\text{Yb}^{3+}$  terminal state. The absence of this line in  $\text{LiYbF}_4$  could therefore be attributed to self-quenching, even at a lower temperature, and is a concentration effect.

<sup>3</sup>D. E. Wortman, S. Kulpa, and C. A. Morrison, *J. Opt. Soc. Am.*, **62** (1972), 604.

<sup>5</sup>D. E. Wortman, *J. Phys. Chem. Solids*, **33** (1972), 311.

<sup>7</sup>N. Karayianis and R. T. Farrar, *J. Chem. Phys.*, **53** (1970), 3436.

TABLE II. Fluorescence recorded for LiYbF<sub>4</sub>, LiTmF<sub>4</sub>, and LiYF<sub>4</sub> crystals doped with rare earths.<sup>a</sup>

Crystal	Crystal temperature	
	300 K	85 K
LiYbF <sub>4</sub> :0.5% Nd <sup>3+</sup> + 0.5% Tm <sup>3+</sup>	Tm <sup>3+</sup> emission with dominant peak at 1.742 $\mu$ m (1.8); Yb <sup>3+</sup> emission centered at 1.03 $\mu$ m (7.5)	Broad Tm <sup>3+</sup> emission near 1.915 $\mu$ m (6.5) and sharp Tm <sup>3+</sup> line at 1.798 $\mu$ m (6.5); Yb <sup>3+</sup> emission centered at 1.02 $\mu$ m (55)
LiTmF <sub>4</sub> :2% Nd <sup>3+</sup>	No measurable emission	No measurable emission
LiYF <sub>4</sub> :2% Nd <sup>3+</sup>	Most intense lines centered at 1.073 $\mu$ m (8) and 1.053 $\mu$ m (37)	Emission similar to that found at 300 K with major lines at 1.073 $\mu$ m (6) and 1.053 $\mu$ m (24)
LiYbF <sub>4</sub> :0.5% Tm <sup>3+</sup>	Tm <sup>3+</sup> emission near most intense line at 1.742 $\mu$ m (2.4); Yb <sup>3+</sup> emission centered at 1.03 $\mu$ m (1.2)	Broad Tm <sup>3+</sup> lines near 1.915 $\mu$ m (4.2) and sharp Tm <sup>3+</sup> line at 1.796 $\mu$ m (4.2); also, Yb <sup>3+</sup> emission centered at 1.02 $\mu$ m (26)
LiYF <sub>4</sub> :2% Yb <sup>3+</sup>	Broad Yb <sup>3+</sup> emission with peaks at 1.016 $\mu$ m (59), 0.996 $\mu$ m (50), and 0.995 $\mu$ m (50)	Sharper Yb <sup>3+</sup> emission with major peaks at 1.02 $\mu$ m (49) and 0.995 $\mu$ m (47)
LiTmF <sub>4</sub>	Broad fluorescence near 1.94 $\mu$ m (two times noise level!) --very weak	Tm <sup>3+</sup> emission with dominant peak at 1.91 $\mu$ m (7)

<sup>a</sup>The wavelengths corresponding to a given transition are given in micrometers, followed by the associated line height in parentheses. The spectrometer slit width in every case was 3.0 mm.



The comparison of absorption data indicates that the crystal field splittings of the  $^4\text{I Nd}^{3+}$  energy levels are nearly the same for  $\text{Nd}^{3+}$  in  $\text{LiYbF}_4$ ,  $\text{LiYF}_4$ , and  $\text{LiTmF}_4$  even though large intensity differences may occur for corresponding transitions in the different crystals. Since, to a rough approximation, the crystal splittings are dependent on the even crystal field parameters and the intensity is dependent on the odd parameters, this change in intensity can be attributed in part to changes in the odd crystal field parameters.<sup>7</sup> It may be attributed also to crystal orientation; however, the even-fold parameters are expected to be nearly those reported<sup>5</sup> for  $\text{Nd}^{3+}$  in  $\text{LiYF}_4$ .

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<sup>5</sup>D. E. Wortman, *J. Phys. Chem. Solids*, **33** (1972), 311.

<sup>7</sup>N. Karayianis and R. T. Farrar, *J. Chem. Phys.*, **53** (1970), 3436.



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